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# HOT ISOSTATIC CONSOLIDATION OF P/M SUPERALLOYS

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## ABSTRACT

The kinetics of powder consolidation, or densification, and the powder morphological changes ocurring during hot isostatic pressing (HIP) are studied as a function of particle size distribution and hold time at HIP temperature for the nickel base superalloy RENE-95. In order to understand the extent of individual powder particle deformation during consolidation and its effect on subsequent prior particle boundaries (PPB), particle size distribution was studied as a variable. Particle size distributions studied include monosized (75-90 um), bimodal ( 75-90 um and 33-35 um) and commercial (<104 um) size distributions. The experimental results of HIP densification kinetics are compared with a newly developed analytical deformation mechanism model for HIP consolidaiton which takes into account the effect of a distribution of particle sizes on the kinetics of densification.

## INTRODUCTION

Mechanisms that can contribute to consolidation during hot isostatic pressing (HIP) of alloy powders include plastic flow above the local yield stress, dislocation creep, superplastic flow, and diffusion. Depending on which mechanisms operate and to what extent during HIP, many powder particles and particle boundaries, which can be sites for the deleterious precipitation of carbides and other particles, can remain undeformed. Discrete particles or particle films at these prior particle boundaries (PPB) in as-HIP superalloys have been shown to impair workability, toughness, ductility, and other mechanical properties and to inhibit grain growth [1-3]. This article reports the initial results, both theoretical and experimental, of a program which examines the kinetics of HIP consolidation of superalloy powders and studies the effects of particle size and size distribution, temperature, pressure and time on the basic mechanisms of consolidation during HIP, as well as the effects of these variables on the extent and morphology of PPB's.

## MATERIALS PROCESSING AND EXPERIMENTAL PROCEDURE

P/M nickel base RENE 95 superalloy powders, whose chemistry is given in Table I, were sieved and classified into three different powder particle size distributions which have been

TABLE I. Nominal Alloy Composition of RENE 95 in Weight Percent

| C | B | Ni | Al | Ti | Cr | Co | Mo | W | Nb | Zr |
|------|------|------|-----|-----|------|-----|-----|-----|-----|------|
| 0.07 | 0.01 | 62.4 | 3.5 | 2.5 | 13.0 | 8.0 | 3.5 | 3.5 | 3.5 | 0.05 |

TABLE II. Percent Theoretical Densities at 103 MPa HIP Pressure

| Particle Distribution | Hold Time (min) | |
| --- | --- | --- |
| | 5 | 180 |
| Monosize | 100 | 100 |
| Bimodal | 100 | 100 |
| Commercial | 100 | 100 |

labeled as monosized (75-90 um), bimodal (80 w/o 75-90 um and 20 w/o 33-35 um) and commercial (<104 um). Powders were canned and then HIP'ed at the temperature of 1120 C for various hold times ranging from 5 minutes to 180 minutes at pressures of either 10.3 MPa or 103 MPa. A one hour ramp schedule was required for each HIP cycle in order that each work piece reach temperature and pressure. The HIP temperature of 1120 C is a typical HIP temperature since it is in the range slightly below the gamma prime solvus for RENE 95, whereby the presence of gamma prime particles retards grain growth during consolidation. The can material was removed mechanically, followed by microstructural observations and density measurements (volume was measured by an air compression pycnometer). Percent theoretical density values were calculated using the density of the commercial powder distribution HIP'ed at 103 MPa for 180 minutes as representing 100% theoretical density.

## EXPERIMENTAL RESULTS

Within experimental error, 100% theoretical density was reached by all three powder particle size distributions within the first five minutes hold time at temperature and 103 MPa HIP pressure, as shown in Table II. Densification occurred predominantly by athermal plastic deformation of the powder particles, with PPB's visible in the microstructures of all three types of consolidates, as shown in Figure 1 (page 3). Increasing hold times to 180 minutes resulted in a coarsening of the gamma prime and an apparent decrease in porosity probably due to lattice and grain boundary diffusion [4,5].

Reducing the applied HIP pressure by a factor of ten resulted in a marked slowing of the densification kinetics for the commercial powder size distribution, as shown in Table III. Powder morphological changes accompanying the increase in densities are represented in Figure 2 (page 3). Note that the initial deformation of the powders within the first 15 minutes at temperature and pressure appears to be primarily by plastic deformation of the smallest powder particles.

TABLE III. Percent Theoretical Densities at 10.3 MPa HIP Pressure

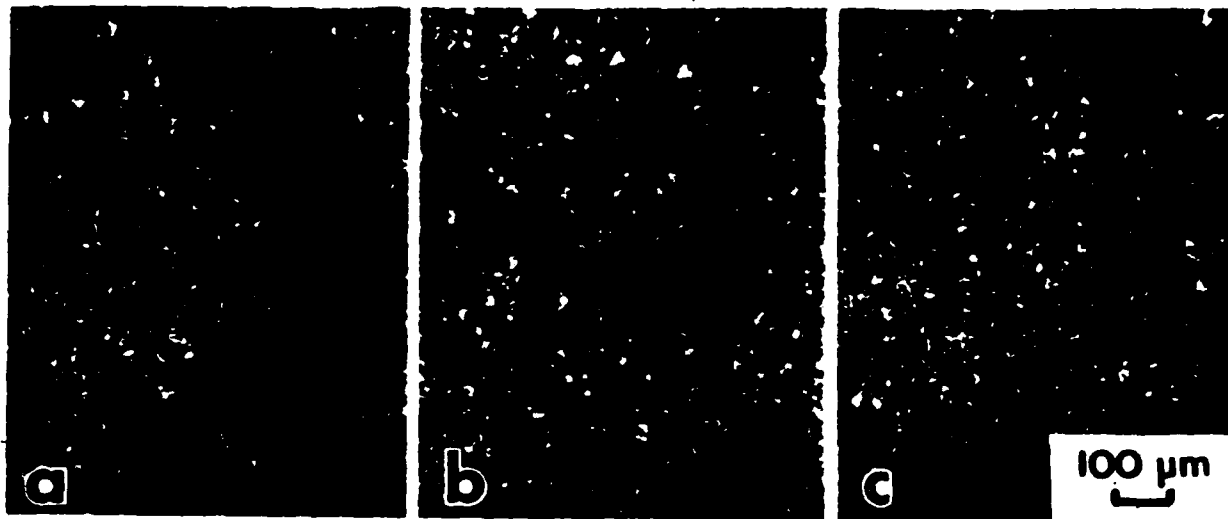| Particle Distribution | Hold Time (min) | | |
| --- | --- | --- | --- |
| | 5 | 15 | 180 |
| Commercial | 92.6 | 96.5 | 99.6 |

FIG. 1. Optical micrographs of HIP'ed RENE 95 powders after a five minute hold time at 103 MPa and 1120 C for a) monosized, b) bimodal, and c) commercial powder size distributions.

## HIP DENSIFICATION MODELLING OF A BIMODAL POWDER DISTRIBUTION

A model has been developed by Nair and Tien [6] to incorporate the influence of particle size distribution on a) the kinetics of densification of metal powders during HIP , and b) the HIP densification mechanism map. In the past, only a monosized distribution of powders were considered [7] and their results are shown as a dashed line in Figure 3 (page 5) for a typical superalloy wherein the regions of different mechanisms of HIP densification are plotted on the normalized scales of relative density, D, and normalized pressure, P/Y, where Y is the material yield strength. It is clear from the figure that at the typical HIP conditions of 103 MPA and 1120 C for RENE 95, densification is almost instantaneous and most of the densification occurs by athermal plastic flow.
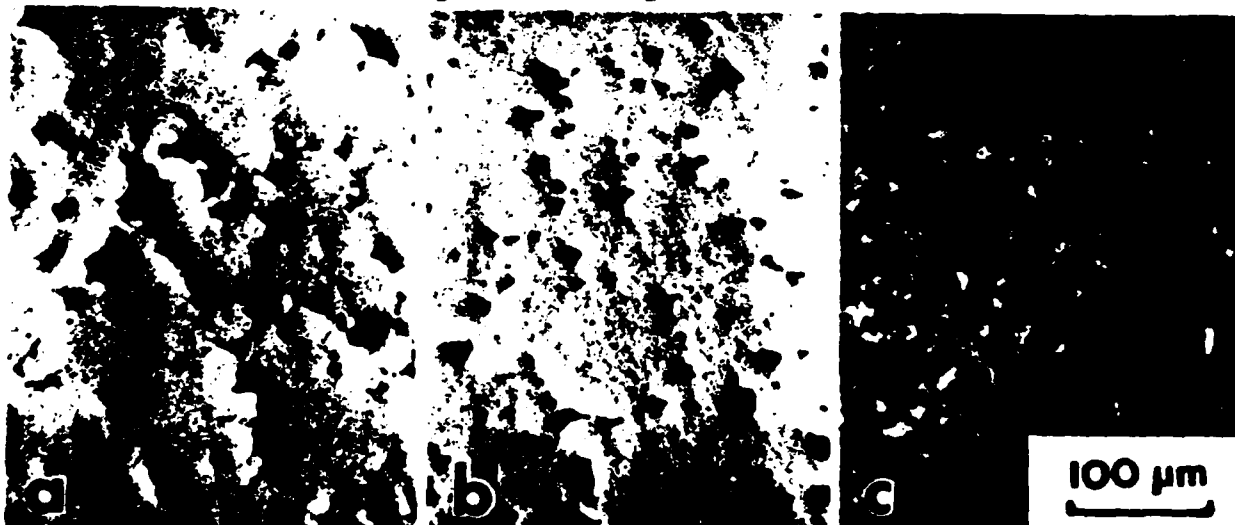


FIG. 2. Optical micrographs of HIP'ed RENE 95 powders (commercial size distribution) showing morphological changes of powders as a function of hold time at 10.3 MPa and 1120 C: a) 5 minutes, b) 15 minutes, and c) 60 minutes.

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How are the results changed when the powders are no longer of uniform size? In order to rederive the HIP densification mechanism map for this case, the new model begins by 1) deriving expressions for the distribution of unequal sized particles in a random mixture and 2) deriving expressions for the relationship between the interparticle contact force and the applied pressure, in other words, by modifying the original Molerus' [8] equation applicable to the monosized particle case.

The results of 1) are obtained in open form using the Percus-Yevik [9] hard-sphere approximation for the molecular structure of liquid alloys as derived by J. L. Lebowitz [10], namely,

$$r\, g_{ij}(r) = \frac{1}{2\pi i} \int_{\delta+i\infty}^{\delta+i\infty} 12(\eta_i \eta_j)^{\frac{1}{2}} G_{ij}(s)\, exp(sr)\, ds \qquad (1)$$

where $g_{ij}(r)$ is the radial distribution function between the ith and jth particle, $\eta_i = \pi\rho_i/6$ where $\rho_i$ is the density of the i-type atom in #/unit volume. The number of atoms at any given distance, r, from a central atom is given by

$$N_{ij}(r) = \rho_j 4\pi r^2 g_{ij}(r) \qquad (2)$$

The final result of 2), which is the modified Molerus Equation for the bimodal case, is

$$\sigma_{app} = (F/4\pi) \left[ (1-\varepsilon-f_2)G_{12}/r_1^3 + (1-\varepsilon-f_1)G_{12}/r_2^3 \right.$$
$$\left. + (1-\varepsilon-f_2)G_{11}/r_1^3 + (1-\varepsilon-f_1)G_{22}/r_2^3 \right] \qquad (3)$$

where $\sigma_{app}$ is the applied stress; F the interparticle contact force; $\varepsilon$ the void volume fraction; $f_1$ and $f_2$ the volume fractions of particles 1 and 2 with respective radii $r_1$ and $r_2$ ; $G_{11}$, $G_{12}$ and $G_{22}$ are the coordination numbers 11, 12 and 22, respectively.

Given the above new formulations described by equations (1), (2) and (3), the new model then proceeds to derive for the bimodal case the density as a function of time using the premise, as in the Arzt model [7], that the densification can be modelled by a fictitious growth of the particles around their centers. The equations involved here are those of mass balance and geometry with the main difference being that the average interparticle stresses are now no longer the same for the two particles.

The final results are shown in Figures 3 and 4 (page 5) for the bimodal case for a typical superalloy at 1120 C. Here particle 1 is the smaller of the two particles with the ratio of particle 2 diameter to particle 1 diameter being approximately 2.3. The points to be noted for the bimodal case in comparison with that for the monosized case are three fold. 1) The two particles do not, in general, deform by the same deformation or densification mechanism. As seen in Figure 3, particle 2 has a substantially larger athermal plastic flow field in comparison with particle 1. For example, at P=Y (the applied stress equals the yield stress) particle 2 deforms almost entirely by athermal plastic flow whereas particle 1 deforms essentially by power law creep. 2) The spread between the two different sized particles

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occurs about that for the monosized particle case in Figure 3, with the boundary for the smaller particle region lying to the right. 3) The final point of importance is shown in Figure 4. This figure indicates that although the smaller particles suffer relatively smaller stresses, they are subjected to much higher levels of plastic strain. The larger the size ratio, the greater the degree of plastic strain suffered by the small particles in comparison with the larger ones.

## DISCUSSION

The almost instantaneous densification of the monosized particles at the typical HIP conditions of 103 MPa and 1120 C is consistent with the resuts of the original Arzt et. al. [7] model. This model indicates, as seen in Figure 3, that most of the densification at these HIP conditions occurs by athermal plastic flow of the powder particles and also predicts that the compacts should be fully densified in the order of minutes, as indeed observed.

The new model of Nair and Tien [6] also predicts that densification is achieved very rapidly at 103 MPa and 1120 C for the case of the bimodal particle size distribution, but the
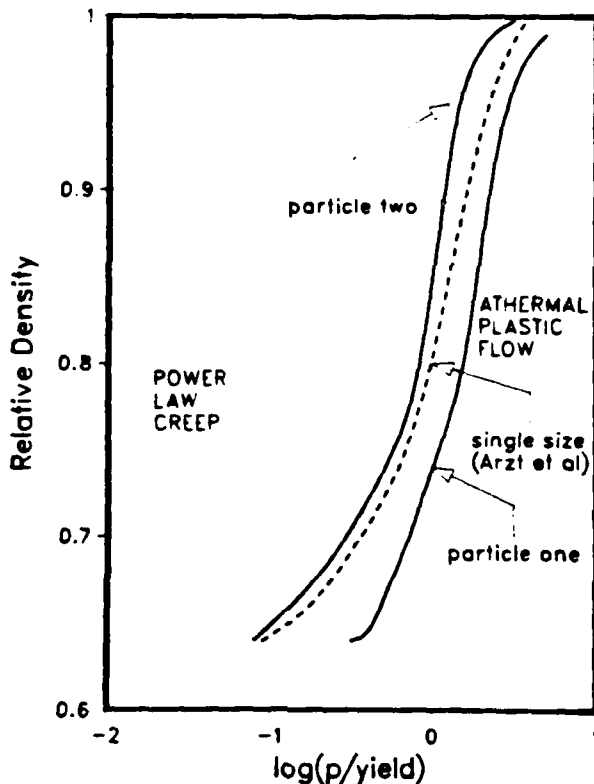


FIG. 3. Calculated HIP densification mechanism map for a superalloy. The dashed line is from Arzt et al. [7] for a single size powder distribution. The solid lines are calculated by Nair and Tien [6] for a bimodal distribution. Particle 1 is the smaller particle.
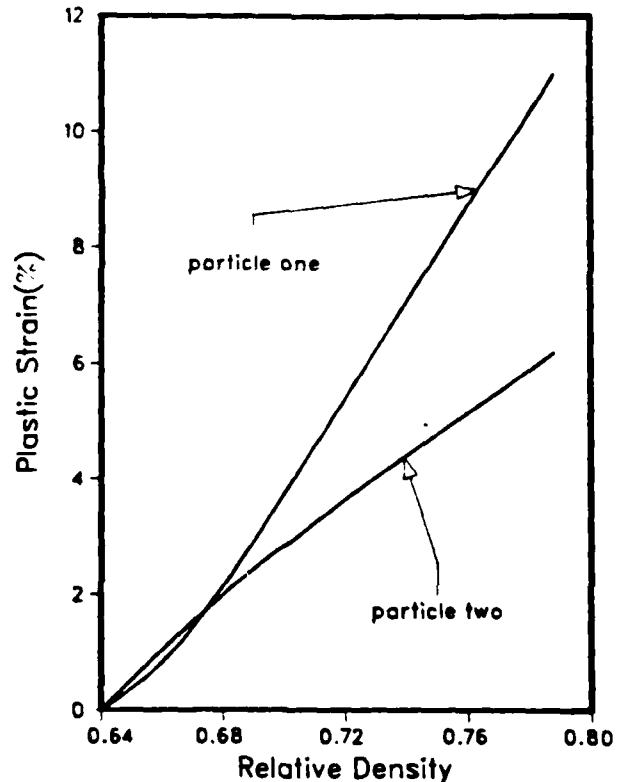
FIG. 4. The plastic strain suffered by particles during HIP for the bimodal particle size distribution as calculated by Nair and Tien [6]. Particle 1, the smaller particle, suffers the larger plastic deformation.

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mechanisms of deformation are predicted to be different from that of the monosized case. Firstly, as indicated in Figures 3 and 4, the smaller particles are predicted to suffer most of the deformation, fully consistent with the results in Figure 2, which shows that the smaller particles are "squashed" in between the large particles which stay relatively undeformed. The new model also predicts, which the present results cannot confirm, that the different size particles do not, in general, deform by the same mechanism of plasic flow.

With respect to prior particle boundaries, these are clearly visible in the fully dense material after BIP at 103 MPa and 1120 C. For both the bimodal and commercial powder size distributions, there appears to be a tendency for the PPB sizes to be skewed to the larger particle sizes. In other words, there is some evidence from the present and previous [11] results that the smaller particles undergo deformation around the larger particles. It is therefore not unreasonable to expect the PPB's to be confined to the larger end of the the particle size spectrum. The new model [6] for BIP densification provides the physical and mechanistic rationale for PPB formation. The smaller particles suffer the greater degree of plastic deformation and leave the larger particles relatively undeformed.

## CONCLUSIONS

It would appear that the BIP parameters of pressure and temperature, where the temperature range is just below the gamma prime solvus, result in component densification achieved almost entirely by time independent plastic deformation of powder particles. The smaller partices in a distribution of particle sizes suffer a higher degree of plastic deformation in comparison with the larger particles, and further, the different sized particles do not, in general, deform by the same mechanism of densification. This result provides a rationale for the formation of PPB's during BIP. Further work needs to be done to extend these results to different temperature and pressure regimes wherein the mechanisms of deformation and densification during BIP are not necessarily dominated by athermal plastic flow and can include also, for example, localized creep and/or superplastic flow of the powder particles.

## ACKNOWLEDGEMENTS

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